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METALS TECHNOLOGY LABORATORIES

REVIEW OF CRYOGENIC TREATMENT ON THE PROPERTIES OF MATERIALS

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ABSTRACT

Cryogenic treatment, the use of low temperature to improve the performance of tool steels started in the 1930. It was as controversial then as it is now as to whether it improves the wear resistance of cutting tools. To metallurgists it is simple transformation of retained austenite to martensite or a further stabilization of retained austenite which could improve wear resistance by 10%. But claims published since 1960 show tremendous results. Even though tool materials have been developed to such an extent that diamond, cubic boron, ceramics and their coatings can improve the performance many folds, the bulk of tool materials are still tool steels. If the life of tool steels can be improved by cryogenic treatment it would benefit the resource industries tremendously. This review briefly examines the history of cryogenic treatment, its claimed benefits, treatment facilities in Canada, processing of tool steels, the effect of cryogenic treatment on tool steels and the potential benefits of cryogenic treatment to the resource industries.

LABORATOIRES DE LA TECHNOLOGIE DES MÉTAUX
RAPPORT MTL 91-4(TR)

EXAMEN DES EFFETS DU TRAITEMENT CRYOGÉNIQUE SUR LES PROPRIÉTÉS DES MATÉRIAUX

par

K.C. Wang

RÉSUMÉ

Le traitement cryogénique, soit l'utilisation de basses températures pour améliorer le rendement des aciers à outils, remonte aux années 1930. Aujourd'hui comme à cette époque, la question de savoir si le traitement améliore la résistance à l'usure des outils de coupe soulève encore une controverse. Pour les métallurgistes, il s'agit d'une simple transformation en martensite de l'austénite retenue ou d'une stabilisation accrue de l'austénite retenue qui peut améliorer de 10 % la résistance à l'usure. Toutefois, les thèses soutenues qui ont été publiées depuis 1960 font état de résultats extraordinaires. Bien que les matériaux servant à la fabrication d'outils soient développés à tel point que le diamant, le bore cubique, la céramique et leurs revêtements permettent d'améliorer sensiblement le comportement, les aciers à outils représentent encore la plus grande partie de ces matériaux. Il serait très avantageux pour les industries extractives de pouvoir améliorer la durée de vie des aciers à outils grâce au traitement cryogénique. Le présent rapport examine l'histoire du traitement cryogénique, ses avantages prétendus, les installation de traitement au Canada, le traitement des aciers à outils, les effets du traitement cryogénique sur les aciers et les avantages éventuels de ce traitement pour les industries extractives.

INTRODUCTION

The use of low temperature treatment to improve the performance of tool steels can be traced back to A. Gulyaev in 1937 (1,2). Subsequently several papers were published by Morris Cohen and his group at MIT on the phase transformation of retained austenite into martensite by the subatmospheric temperature treatment (3-15) and his contemporary metallurgists (16-22). The terms subatmospheric temperature, subzero, cold, and cryogenic treatment all refer to the treatment at temperatures of -80°C or lower to reduce the retained austenite to maintain dimensional stability, hence it is the stabilizing treatment for tool steels with retained austenite. In 1966, Barron of Louisiana Tech University and a heat-treating company, Materials Improvement, made claims about the improved wear properties obtained by cold treatment. A lot of claims were published, mostly in the trade journals (22-38) and in Russian journals as well (39-43). These include improvement in wear resistance of high speed and carbide tools, stabilization of the dimensions of aluminum parts, extension of life in copper resistance-welding electrodes. More amazing claims include piano strings that stay tuned longer, nylon hose that does not run easily, golf balls that fly farther, etc. Some of the claims with known company names are shown in Table 1 and Barron' results are shown in Table 2.

Our current interest is to improve the wear resistance of tools used in the mining, forestry, wood processing and general manufacturing industries. The materials concerned are tool steels and carbides.

COLD/CRYOGENIC TREATMENT FACILITIES IN CANADA

Telephone interviews with Canadian companies working in the cold/cryogenic treatment field were conducted. Most of the cold-treatment function are part of heat-treatment plants that use cryogenic treatment as specified by the customers according to heat-treatment manuals or military specifications as listed in Table 3 (56). Others have developed more advanced techniques to suit the requirements of individual applications. Most of the applications are for die materials where dimensional stability is critical. Others use cryogenic treatment to reduce or to stabilize retained austenite. There are also companies doing cryogenic treatment mainly to improve the performance of cutting tools and welding tips. Tables 4 and 5 list Canadian companies with cryogenic/cold treatment facilities.

AUSTENITE TRANSFORMATION

For most tool steels the carbon and alloy contents are high enough that the pearlitic and bainitic transformations can be suppressed by rapid quenching to below the knee temperature on

the TTT curves. To transform austenite to martensite the alloys have to be cooled below their M_S temperatures, which are influenced by the composition of the austenite. The Ms temperatures for tool steels are shown in Table 6 based on several models and alloy compositions (11,44,45). These Ms temperatures are approximate because the austenite compositions may be significantly different from the alloy composition however, they will provide some guidance. The alloys under consideration, M2, D2 and D3 all have Ms temperatures close to room temperature or lower. This indicates that Mf temperatures will be even lower, hence there will be retained austenite during initial quenching to room temperature. Because austenite has a lower lattice volume than martensite, it expands during the austenite-martensite transformation and generates elastic residual stress during quenching. The retained austenite is unstable and can transform under stress to generate extra tensile stress in the matrix, causing excessive tool wear. A tempering treatment is used to reduce retained austenite, relieve residual stress and to stabilize the tool dimensions. For cutting tools with high alloy concentrations, the tempering temperature is in the 450-600°C range. At this temperature, secondary alloy carbides will be precipitated. The alloy and carbon concentrations in the remaining austenite are reduced, and hence, the Ms temperature will be raised, causing the remaining austenite to transform to martensite during cooling from the tempering temperature and generating additional stress. To relieve this elastic stress and to reduce retained austenite further, double temper, triple temper even quadruple temper are used to reduce retained austenite and to relieve the residual stress.

CRYOGENIC TREATMENT

Instead of a high-temperature temper treatment, a low-temperature treatment may be used with the same objective to reduce retained austenite, relieve residual stress, stabilize tool dimensions and to extend the tool life. The materials, mostly high-alloy tool steels or bearing steels, have the following characteristics after cryogenic treatment:

1. higher ductility (3).
2. higher elastic constant and higher maximum stress (3,12) as shown in Fig. 1.
3. higher fracture stress and fracture strain hence higher total fracture energy (12) shown in Fig. 2 and 3.
4. better dimensional stability for gage applications (5-7,10).
5. Better wear resistance (21-43).

Even though cryogenic treatment has the above mentioned advantages, most heat-treatments are used only to reduce retained austenite for dies and components since excessive retained austenite may cause premature failure due to dimensional change. The reported improvements in wear resistance of tools are not conclusive, not only in north America but also shown in Russia.

Zhmud stated that shock cooling was tested in Russia in 1977 at 209 plants with finished tools made from 22 types of high speed steels (39). These tools included drills of 0.3-26 mm in diameter, milling cutters, taps and dies, countersinks, broaches, hacksaw blades, files, cutting pliers, scissors, knives, disk saws, saw segments, clamps, burnishing tools, combs, gear cutters, draw plates, dies and punches. The results are listed below.

Results obtained	No. of plants	(%)
Service life increased > 2 times	98	(50)
Service life increased in some cases but had no effect in others	38	(18)
Service life of some tools increased but others decreased	7	(3)
No effect	50	(24)
Negative results	11	(5)

The results were inconsistent, but 70% had a positive effect. Another test with R6M3 steel broaches (6% W, 3% Mo) showed the following results:

Processing procedure	Tool life (number of hole broached)
Standard (quenching + double tempering + machining)	600
Quenching + double tempering + machining + cold treatment	900
Quenching + single tempering + machining + cold treatment	2000
Quenching + machining + cold treatment	2500

In this test, the broach, cold treated directly after quenching, had the best performance.

From Zhmud's results the wear resistance of cryogenic-treated specimens directly after quenching is far superior to normal temper treatment to bring out alloy carbides during secondary hardening. In another claim by Hamilton, one drill after cryogenic treatment could drill over 100 holes in aluminum-40% SiC composites whereas without cryogenic treatment three drills were needed to drill one hole. Although these claims have to be repeated under controlled conditions to be valid, it certainly indicates that tool steels after cryogenic treatment possess properties that might be overlooked.

The effect of cryogenic treatment on the wear resistance of tool steels is a controversial subject even though metallurgists agree the effect is caused by the transformation of retained austenite to martensite. Why cryogenic treatment works on some materials and not on other materials and what we can do at MTL to solve this puzzle need to be defined. We have to understand the processing steps for tool steels first. A few related publications are listed in References 44 to 56 and in related texts.

PROCESSING OF TOOL STEELS

Tool steels are generally sold in the annealed condition. After machining, tools are austenitized through preheating stages and held at the austenitizing temperature for a certain time. The temperature and time at temperature are critical as dissolution of primary carbides into austenite and grain growth are both proceeding at the same time. The M_S temperature is related to the carbon and alloy concentration in the austenite and not with the total carbon and alloy content in the alloy. Carbon in carbides has no effect on the martensitic transformation.

The austenitizing temperature and time can be controlled to give desired structures and properties. Higher temperature and a longer time will yield a higher carbon and alloy concentration in the austenite, hence, a lower Ms temperature and larger grain size. Consequently, there will be more retained austenite after quenching to room temperature. To avoid cracking, a lower austenitizing temperature, quenching to an intermediate temperature above Ms then cooling to room temperature, or a lower quenching rate can all be used to reduce the possibility of cracking.

To relieve the stress generated during quenching and preventing retained austenite from stabilizing at room temperature, tool materials should be tempered immediately after reaching room temperature or slightly higher than room temperature. The tempering temperature ranges from 160°C to 500°C depending on the final hardness and toughness required. Secondary hardening occurs when the increase in hardness caused by the precipitation of alloy carbides and further martensitic transformation during cooling exceeds the lowering of hardness by relieving the stress generated during quenching. To reduce retained austenite and to temper martensite formed during cooling from tempering temperature, multiple temper treatments, up to a quadruple temper, are commonly used.

While tempering is necessary after quenching, a stabilizing treatment is optional. Stabilizing treatment is generally required for intricate dies with abrupt changes in section size. Parts cool down further to -85°C or -196°C after quenching to reduce retained austenite and to improve dimensional stability. Tempering is required immediately after reaching room temperature (47).

DISCUSSION

Cryogenic treatment has been part of the heat treatment of tool steels for many years. The purpose of the treatment is to stabilize the dimensions of intricate dies by converting retained austenite to martensite and to lower the stress-induced martensitic temperature Md to below room temperature, so that martensite will not form under stress. The claims of the improvement of wear resistance are voluminous yet so controversial that an explanation must exist.

The structures of quenched tool steels both after the temper treatment at secondary hardening temperature and after cryogenic treatment have common features in undissolved primary carbides and fine grain structures with carbides preventing grain growth under controlled conditions. The different features due to treatments are tabulated below:

Tempered	Cold treated
1. Additional carbides precipitated during tempering.	1. Fine clusters in the 5 to 10 nm range (29).
2. Fully tempered original martensite plus additional martensite formed during cooling from tempering temperature.	2. Partially tempered.martensite only
3. Tensile residual stress from newly-formed martensite.	3. Relieved tensile stress or compressive residual stress from tempered martensite.
4. Hardness could be higher or lower depending on the hardening and tempering temperature.	4. Hardness could be higher or lower depending on the amount of retained austenite.
5. Less brittle than quenched state.	5. 2 to 4 times more ductile after heated to tempering temperature (3).

It is well known that hardness is directly related to abrasive wear resistance according to the equation (49):

$$V = \frac{\tan\theta}{\pi}\,\frac{PL}{H}$$

Where V is wear volume, P is normal load, L is sliding distance, H is hardness of abraded material, θ is attack angle and $\tan\theta$ is a weighted average of the $\tan\theta$ values of all contacting asperities.

$$W^{-1} = \frac{1}{V} = \frac{\pi}{\tan\theta}\,\frac{H}{PL}$$

Wear resistance, W^{-1}, is the reciprocal of weight loss, which is directly proportional to hardness of the abraded material.

This is the simplest form of relating hardness to abrasive wear resistance. Barron showed after cryogenic treatment that wear resistance increases but the hardness does not change

appreciably. There are proposed mechanisms for the increasing wear resistance, such as precipitation of fine carbides, compressive residual stress and reduction of retained austenite.

There could be other mechanisms operating. Kurdjumov showed that at low temperatures martensitic transformation can proceed isothermally and that the transformation rate depends on temperature as shown in Fig. 4. From liquid nitrogen temperature to room temperature and to 200°C alloy atoms may not be able to diffuse far but carbon atoms were observed to form clusters at -160°C. And at 100°C carbon started to diffuse out from highly strained HCP or BCT martensitic lattices to reduce the carbon content in martensite and eventually become the BCC type cementite. The hardness of the matrix tempered at 150° to 200°C does not decrease much but the strain could be lower.

In many claims, cryogenically-treated tools need to be taken much less during regrinding, about one third the amount of regularly tempered tools. This effect may be due to the compressive stress in the matrix and the finely precipitated secondary carbides which limit the crack propagation to short distance. This compressive residual stress could also protect the carbides from cracking and pull-out.

Why are cryogenic treatments so controversial? It is evident that only those materials that have retained austenite can have their abrasive wear resistance improved by a cryogenic treatment. Those materials that will not improve as a result of cryogenic treatment are as follows:

1. materials that do not have retained austenite.
2. materials that have already been cryogenically treated.
3. materials that have retained austenite, but because of stabilizing treatment the M_S temperature has dropped below the cryogenic treatment temperature.
4. materials that have M_S temperature lower than the commonly used cryogenic treatment temperature. In this case, further cooling to a lower temperature (such as liquid argon temperature) may work but may not be economically practical.

CONCLUSIONS

Cryogenic treatment is a controversial subject, but it is being practised by many shops. At present, carbides, ceramics and superhard materials are available for tool applications. The bulk tools are still based on tool steels. If the claims about cryogenic treatment can be proven, the economic benefit to the Canadian industry can be tremendous, so that the effect should be worth investigating.

REFERENCES

1. A. Gulyaev, Improved heat treatment of high speed steel to improve cutting quality. Metallurg, Vol. 12, 1937, pp 65-77.

2. A. Gulyaev, Transformation of retained austenite at sub-zero temperature with high-alloy steels. Metallurg, Vol. 14, 1939, pp 64-71.

3. P. Gordon and M. Cohen, The transformation of retained austenite in high speed steel at subatmospheric temperatures, ASM Transactions. Vol.30, 1942. pp 569-591.

4. P. Gordon, M. Cohen and R.S. Rose, Effect of quenching-bath temperature on the tempering of high speed steel, ASM Transactions. Vol.32, 1944. pp 411-454.

5. S.G. Fletcher and M. Cohen, The dimensional stability of steel. Part I - Subatmospheric transformation of retained austenite. ASM Transactions, Vol. 34, 1945, pp 216-249.

6. S.G. Fletcher, B.L. Averbach and M. Cohen, The dimensional stability of steel. Part II - Further experiments on subatmospheric transformation. ASM Transactions, Vol. 40, 1948, pp 703-727.

7. B.L. Averbach, M. Cohen and S.G. Fletcher, The dimensional stability of steel. Part III - Decomposition of martensite and austenite at room temperature. ASM Transactions, Vol. 40, 1948, pp 728-757.

8. B.L. Averbach and M. Cohen, The isothermal decomposition of martensite and retained austenite. ASM Transactions, Vol.41, 1949. pp 1024-1060.

9. W.J. Harris, Jr. and M. Cohen, Stabilization of the austenite-martensite transformation. Trans. AIME, Vol.183, 1949. pp 447-470.

10. B.S. Lement, B.L. Averbach and M. Cohen, The dimensional stability of steel. Part IV - Tool steels. ASM Transactions, Vol. 41, 1949, pp 1061-1087.

11. M. Cohen, Retained austenite (1948 Edward DeMille Campbell Memorial Lecture). ASM Transactions, Vol.41, 1949. pp 35-94.

12. L.S. Castleman, B.L. Averbach and M. Cohen, Effect of retained austenite upon mechanical properties. ASM Transactions, Vol 44, 1952, pp 240-263.

13. F.E. Werner, B.L. Averbach and M. Cohen, The tempering of iron-carbon martensite crystals. Trans. ASM, Vol.49, 1957. pp 823-841.

14. P.G. Winchell and M. Cohen, The strength of martensite. Trans. ASM, Vol. 55, 1962. pp 347-361.

15. M. Cohen, Operational nucleation in martensite transformation. Metall. Trans. ASM, Vol.3, May 1972. pp 1095-1098.

16. L.E. Gippert and G.M. Butler, Jr., Changes in size and toughness of high carbon-high chromium steels due to subzero treatments. Vol. 39, 1947. pp 549-568.

17. G.A. Roberts and J.P. Gill, Some effects of sub-zero cooling on the tempering of high speed steel, The iron age, March 23, 1944, pp 53-56.

18. S.M. DePoy, Sub-zero treatment of high speed steels, The iron age, April 13, 1944, pp 52-55.

19. J.G. Morrison, An appraisal of subzero hardening of high speed steel, Iron Age, July 26, 1945, pp 54-60, August 2, 1945, pp 64-70.

20. R.A. Kennedy, A study of subzero treatment applied to molybdenum-tungsten high speed steel, ASM Transaction, 1944, pp 250-309.

21. G.V. Luerssen and O.V. Green, The cold treatment of certain alloy steels, ASM Transactions. Vol. 2, 1932. pp 501-552.

22. A.H. Grobe and G.A. Roberts, Unnotched impact strength of high speed steels. ASM Transactions, Vol. 42, 1950. pp 686-719.

23. R.F. Barron, Cryogenics--Do temperatures below -120 F help?, Heat Treating, June 1974, pp 3-5.

24. R.F. Barron and C.R. Mulhern, Cryogenic treatment of AISI-T8 ANS C1045 steels. Advances in cryogenic engineering-materials, Vol. 26, 1980. pp 171-179.

25. R.F. Barron, Cryogenic treatment of metals to improve wear resistance, Cryogenics, Vol 22, No.5, Aug. 1982, pp 409-413.

26. R.F. Barron and R.H. Thompson, Effect of cryogenic treatment on corrosion resistance, Cryogenic engineering conference, Los Angeles, July, 1989.

27. Frozen Tools, Popular Science, June 1987, pp 64.

28. R.F. Barron, Cryogenic treatment produces cost savings for slitter knifes. Tappi, Vol. 57, No.5. May 1974. pp 137-139.

29. R.F. Barron, Effect of cryogenic treatment on lathe tool wear. Proceedings of the XIII international congress - Progress in the refrigeration science and technology, 1973. pp 529-534.

30. Ultra-cold treatment up heavy duty tool wear, Iron Age, Feb 11, 1971. p 58.

31. R.A. Wilson, Industry warms up to supercold. Iron Age, Aug. 26, 1971. pp 55-56.

32. R.F. Barron, Yes, cryogenic treatment can save you money! Tappi, Vol. 57, No.5. May 1974. pp 35-40.

33. P.C. Miller, Cryogenics: Solves heat-treat problems. Tooling and Production. Vol. 45, Feb. 18, 1980. pp 82-86.

34. R. Frey, Cryogenic treatment improves properties of drills and P/M parts. Industrial heating. September, 1983. pp 21-23.

35. L.V. Leonard, Enhancing metals properties with supercold: Fact or farce? Mater. Eng., Cleveland, Vol.102, July 1985, pp 29-32.

36. J.A. Vaccari, Deep freeze improves products. American machinist and automated manufacturing, Mar. 1986. pp 90-92.

37. R.F. Barron, Abrasive wear tests on metals with cryogenic treatments. Apr. 1981.

38. R.F. Barron, How cryogenic treatment controls wear. Presented to Beloit corporation. Oct. 1973.

39. E.S. Zhmud, Improved tool life after shock cooling, Metal Science and Heat Treatment, No. 10, Oct. 1980, pp 701-703

40. S.V. Dianov, V.A. Radchenko, V.A. Rudman, and V.P. Severinov, Effect of aging before repeated cold treatment on the mechanical properties of steel N26T. Metal science and heat treatment. V.22, No. 5 & 6, May/June 1980. pp 454-457.

41. A.N. Popandopulo, L..T. Zhukova, and A.G. Yaroshenko, Effect of cooling rate in liquid nitrogen on the transformation rate of retained austenite in steel R6M5. Metal science and heat treatment. V.25, No. 5 & 6, May/June 1983. pp 334-337.

42. A.M. Rogalev, T.I. Zenchenko, and Z.Kh. Ibragimov, Mechanical properties of die steels after low-temperature mechanical treatment. Metal science and heat treatment. V.26, No. 5 & 6, May/June 1984. pp 634-637.

43. I.A. Tananko, V.V. Belozerov, and A.I. Makhatilova, Fracture of high-carbon hardening martensite on the change in it during low-temperature tempering. Metal science and heat treatment. V.26, No. 5 & 6, May/June 1984. pp 713-716.

44. W. Steven and A.G. Haynes, The temperature of formation of martensite and bainite in low-alloy steels-some effects of chemical composition. JISI, Vol.183, 1956. pp 349-359.

45. K.W. Andrews, Empirical formulae for the culculation of some transformation temperatures, JISI, Vol.203, 1965. pp 721-727.

46. P.M. Underwiser, H.E. Boyer and J.E. Kubbs, Heat treater' guide: standard practices and procedures for steel. ASM, 1982.

47. Rees Llwellyn, private communication, Oct. 1989.

48. E. Rabinowics, Friction and Wear of Materials, Wiley , New York, 1965, p 168.

49. G.V. Kurdjumov, Phenomena occurring in the quenching and tempering of steels. JISI, May, 1960. pp 26-48.

50. K.J. Irvine, D.J. Crowe and F.B. Pickering, The physical metallurgy of 12% chromium steels. JISI, Aug. 1960. pp 386-405.

51. H.W. Paxton and J.B. Austin, Historical account of the contribution of E.C. Bain, Metall. Trans. ASM, Vol.3, May 1972. pp 1035-1042.

52. G.R. Speich and W.C. Leslie, Tempering of steel. Metall. Trans. ASM, Vol.3, May 1972. pp 1043-1054

53. G.V. Kurdjumov and A.G. Khachaturyan, Phenomena of carbon atom redistribution in martensite. Metall. Trans. ASM, Vol.3, May 1972. pp 1069-76.

54. R.W.K. Honeycombe and F.B. Pickering, Ferrite and bainite in alloy steels. Metall. Trans. ASM, Vol.3, May 1972. pp 1099-1112.

55. J.S. Bowles and C.M. Wayman, The Bain strain, lattice correspondences, and deformations related to martensitic transformation. Metall. Trans. ASM, Vol.3, May 1972. pp 1113-1121.

56. Heat Treater's Guide, ASM, 1982.

RELATED TEXTS

1. G.A. Roberts, J.C. Hamaker & A.R. Johnson, Tool Steels, 3rd ed., ASM, 1960.

2. G.A. Roberts & R.A. Cary, Tool Steels, 4th ed., ASM, 1980.

3. M.A. Grossmann and E.C. Bain, Principles of Heat Treatment, 5th Ed. ASM, 1964.

4. E.C. Bain and H.W. Paxton, Alloy Elements in Steel, 2nd ed., 3rd rev. ASM, 1966.

5. Phase Transformations, ASM, 1969.

6. Transformation in Metals, Shewmon, 1969.

7. G.B. Rothenberg, Specialty Steels-Recent Developments, 1977.

8. Zenji Nishiyama, Martensitic Transformation, Academic Press, 1978.

9. R.W.K. Honeycombe, Steels-Microstructure and Properties, 1980.

10. Geoffrey Hoyle, High Speed Steels, 1980.

11. N.R. Comins and J.B. Clark, Specialty Steels and Hard Materials, 1982.

12. International Conference on Martensitic Transformations, Leuven (Belgium), Aug 8-12, 1982. Journal de Physique Colloques, 1982.

13. Antolovich, Ritchie and Gerberich Mechanical Properties and Phase Transformations in Engineering Materials- Earl Parker Symposium on Structure Property relationships, 1986.

14. G. Krauss, Steels: Heat Treatment and Processing Principles, ASM, 1989.

Table 1. Test results of regular vs cryogenically treated tools.

Company	Year	Component	Tool Material	Used on	Regular	Cryo	Ratio
Baird	1986	Punch		A1355T6	68 103	121 601	1.79
Baird	1986	Endmill		A1355T6	12	160	13.33
Baird	1986	Endmill		A1355T6	50	160	3.20
Baird	1986	Endmill		A1355T6	75	160	2.13
Baird	1986	Endmill		A1355T6	30	160	5.33
Baird	1986	Endmill		A1355T6	25	160	6.40
Baird	1986	Endmill		A1355T6	30	160	5.33
Baird	1986	Keycutter		A1355T6	35	160	4.57
Baird	1986	Drill		A1355T6	15	160	10.67
Baird	1986	Drill		A1355T6	15	160	10.67
Baird	1986	Drill		Acrylic	15	160	10.67
Comet	1984	Blade			5	120	24.00
Raytheon	1984	Broach			20	90	4.50
Raytheon	1984	Mandrel			1 586	7 107	4.48
Varian	1984	Endmill			20.76	31.56	1.52
Varian	1984	Endmill			15.88	22.95	1.45
Varian	1984	Endmill			15	25.25	1.68
Varian	1984	Endmill			90	126.25	1.40
Varian	1984	Drill			27	36.07	1.34
Varian	1984	Drill			45	252.5	5.61
Conntech	1984	Cutter			2	5	2.50
National Automatic	1983	Milling cutter			1 000	1 450	1.45
Horst	1983	Drill			50	100	2.00
Horst	1983	Cut-off blade			40	3 900	97.50
Mature Engine	1983	Spline cutter	T15		4	13	3.25
Mature Engine	1983	Spline cutter	M2	A286			1
Mature Engine	1983	Spline cutter	APS 30	INCO-716	6	18	3
Danlyn	1982	Drill			123	1 025	8.33
Hamilton	1982	Spade drill	M2 or M10	Mall C.I.	1	7	7.00
Hamilton	1983	Lathe	Carbide		4	10	2.50
G.E.	1982	Drill		Al-40% SiC			
Ciba-Geigy	1981	Punch	S5	Tablets	6 000 000	80 000 000	13.33
Hamilton	1981	Milling cutter	TRW C3 carbide	Fibreglass	30	900	30.00
NEP	1988	Knives	D2	Vinyl/TiO_2	4 wks	3 mo	3.00
Pratt-Whitney		Welding Electrodes	Cu				
Hawthorne Metals		Ditto	Cu		2 wks	6 wks	3.00
Hqwthorne Metals		Progressive die			40 000*	250 000*	6.25
Wellman		Blanking		4140&1095	1 000	2 000	2.00
Chrysler		Broach		C1020	1 810	8 602	4.75
Chrysler		Broach		Connecting rod	1 500	8 600	5.73
Devlieg		Milling	C1018		3	14	4.67
Boise		Cut-off blade	AMT-38		60	928	15.47
Berlin Press		Trimmer blade			28 h	140 h	5.00

* hits

Table 2. Wear test and hardness of heat-treated and crygenically treated materials.

Materials	Wear Resistance Rw			Ratio		Hardness Hv		
	1*	2*	3*	2*/1*	3*/1	1*	2*	3*
P20	6.252	7.695	6.076	1.231	0.972	347	339	343
01	514.3	1139.8	1399.2	2.216	2.721	801	801	801
A2	48.76	47.9	54.41	0.982	1.116	697	709	697
A6	2165	1151	2094	0.532	0.967	747	760	801
A10	1035	2386	2738	2.305	2.645	653	653	653
D2	199	630	1628	3.166	8.181	697	709	697
H13	30.20	49.73	63.25	1.646	2.094	512	505	512
T1	1613	2287	2843	1.418	1.763	801	773	787
T2	5751	5338	4142	0.928	0.720	773	721	760
M1	1748	1129	776	0.646	0.444	801	831	831
M2	3900	4388	4698	1.125	1.205	801	801	816
S7	5.13	12.38	13.38	2.413	2.608	653	632	643
SS303	14.3	15.06	15.84	1.053	1.108	155	150	151
SS430	11.14	12.94	13.35	1.162	1.198	261	238	240
SS440	1447	1853	1763	1.281	1.218	632	632	632
CPM10V	5713	5458	7635	0.939	1.313	831	831	831
AQS MDI	2753	3659	2654	0.966	0.964	697	721	721
8620	8.76	9.08	9.77	1.037	1.115	760	773	773
1020	5.01	4.92	4.87	0.982	0.972	787	747	787

Remarks

1. 1* normally heat treated
2. 2* heat treated then cryogenically treated at -80°C
3. 3* heat treated then cryogenically treated at -196°C
4. Aluminum oxide grinding wheel
5. Surface speed 478 mm/s
6. Normal force 431 N
7. Tabor type test with 12.7 mm diameter x 63.5 mm specimen

Table 3. Cryogenic treatment for tool and martensitic stainless steel.

Materials	Cryogenic treatment		Temperature (°C)
	Required	Temperature (°C)	
W1, W2, W5	No		175 to 345
S1	Optional	-100 to -195	205 to 650
S2, S5	Optional	-100 to -195	175 to 425
S6	Optional	-100 to -195	205 to 315
S7	Optional	-100 to -195	205 to 620
O1, O2	Optional	-100 to -195	175 to 260
O6	Optional	-100 to -195	175 to 315
O7	Optional	-100 to -195	175 to 290
A2, A3, A7	Optional	-85	175 to 540
A4, A10	Optional	-85	175 to 425
A6	Optional	-85	150 to 426
A8	Optional	-85	175 to 595
A9	Optional	-85	510 to 620
D2, D3, D4	Optional	-85	205 to 540
D5	Optional	-85	205 to 540
D7	Optional	-85	175 to 540
L2, L6	No		175 to 540
P2, P3, P5	No		175 to 260
P4	Optional	-85	175 to 480
P6	Optional	-100 to -195	175 to 230
P20, P21	No		
H10, H11, H12, H13	Optional	-100 to -195	540 to 650
H14, H19, H21, H22	Optional	-100 to -195	540 to 650
H23, H24, H25, H42	Optional	-100 to -195	540 to 650
T1, T2, T4, T5, T6	Optional	-100 to -195	540 to 590
T8	Optional	-100 to -195	540 to 590
T15	Optional	-100 to -195	540 to 650
M1, M2, M3, M4, M6	Optional	-100 to -195	540 to 590
M7, M10, M30, M33	Optional	-100 to -195	540 to 590
M34, M36	Optional	-100 to -195	540 to 590
M42, M43	Optional	-100 to -195	510 to 595
M44	Optional	-100 to -195	540 to 625
M46	Optional	-100 to -195	525 to 565
Martensitic stainless steel			
403, 410, 414, 416	Yes	-100 to -195	205 to 760
420	Yes	-74	205 to 370
422	Yes	-74	650
430	Yes	-74	230 to 605
440	Yes	-74	150 to 370

Note: Tempering between 370 to 565 not recommended for martensitic stainless steel.

Table 4. Cold/cryogenic treatment facilities in Canada.

Company	Minimum temperature (°C)	Freezer size (in.)	Main usage
Allied Heat Treating Ltd.	-196	30 x 24 x 24	Tool & HS steel, Al parts
B & W Heat Treating Ltd.	-84		Dies, taps
Davidson Heat Treat Inc.	-84		SS, Hi-Ni alloys
H & S Heat Treat Inc.	-196	26 x 26 x 60	Tools, mining striker bars, forgings
Ipsenlab of Canada Ltd.	-196	22 x 28 x 48	Al
McLeod & Norquay Ltd.	-84	60 (diam) x 36	Tool steels
Metcor Inc.	-84		Military parts
Vac-Aero International Inc.*	-84		
Vac-Aero International Inc.**	-84		SS, tool steels
F.C. Hume & Co.	-196	≤72 x 72 x 192	Cu tips, forestry tools
Applied Cryogenics of Canada Inc.	-196	17 diam x 36	Just start up

* Oakville, Ont.
** St. Laurent, P.Q.

Table 5. Locations of cold/cryogenic treatment facilities in Canada

Company	Location	Telephone (Fax)	Contact
Allied Heat Treating Ltd.	32 Penn Dr., Weston, ON	416-749-5555 (416-742-4263)	Mr. Price
B & W Heat Treating Ltd.	Kitchener, ON	519-748-5284	Claire Benn
Davidson Heat Treat Inc.	694 Drent Way, Annacis Island, New Westminster, BC V5M 5P8	604-525-0241 (604-525-7535)	John Davidson
H & S Heat Treat Inc.	South St. N., Port Robinson, ON	416-384-9355	John McCauley
Ipsenlab of Canada Ltd.	20-7 Bermondsey Rd., Toronto, ON M4B 1Z7	416-757-3233	Dave Kernoghan
McLeod & Norquay Ltd.	520 Raymur Ave., BC, V6A 3L2	604-255-9381	John McLeod
Metcor Inc.	10600 Secant St., Angou Island, Montreal, PQ	514-353-1500	
Vac-Aero International Inc.	Oakville, ON	416-827-4171	
Vac-Aero International Inc.	7450 Verite, St. Laurent, PQ	514-334-4240	
F.C. Hume & Co.	2100 Drummond, Montreal, PQ H3G 1X1	514-842-2991	Pierre Ledilicocq
Applied Cryogenics of Canada Inc.	119 McMaster, Ajax, ON L1S 2E6	416-683-8251 (416-683-4251)	Glen Paul

Table 6. Ms temperature of tool steels.

Alloy		Nominal composition (%)									Ms Temperature (°C)		
No	Name	C	Mn	Si	Cr	Ni	V	W	Mo	Co	S & H	Andrews	Cohen
549	H26	0.50	0.28	0.28	4.13	–	1.00	18.13	–	–	244.6	269.0	37.6
550	H15	0.35	0.25	0.38	3.75	–	0.75	1.00	6.00	–	197.1	293.0	214.5
551	H15	0.50	0.25	0.50	4.25	–	0.75	1.00	5.75	–	122.8	225.4	159.9
552	H43	0.58	0.28	0.30	4.13	–	2.00	–	8.13	–	35.9	174.2	124.4
553	H42	0.63	0.28	0.33	4.13	–	1.98	6.13	5.00	–	77.9	176.5	75.8
554	H41	0.68	0.28	0.33	3.75	–	1.15	1.75	8.60	–	-14.9	133.0	77.9
610	T1	0.73	0.25	0.30	4.43	–	1.10	18.00	0.70	–	121.8	167.4	-36.9
611	T2	0.85	0.30	0.30	4.13	–	2.10	18.13	0.63	–	64.8	115.6	-75.2
614	T3	1.09	0.25	0.25	4.13	–	3.10	18.25	0.80	–	-50.9	14.4	-148.2
615	T9	1.25	0.25	0.25	4.00	–	4.00	18.25	0.75	–	-123.5	-51.4	-192.8
616	T7	0.73	0.25	0.25	4.75	–	1.75	14.00	–	–	126.0	165.1	2.1
620	T4	0.75	0.25	0.30	4.13	–	1.00	18.75	0.70	5.00	112.3	158.9	-23.2
621	T5	0.80	0.30	0.30	4.38	–	2.10	18.75	0.83	8.25	80.0	132.3	-28.7
622	T6	0.80	0.30	0.30	4.38	–	2.30	19.75	0.70	12.00	82.7	133.2	-17.4
623	T15	1.55	0.28	0.28	4.38	–	4.88	12.38	0.50	5.00	-267.9	-181.9	-196.6
624	T8	0.80	0.30	0.30	4.13	–	2.10	14.00	0.75	5.00	85.9	135.9	12.3
630	M1	0.83	0.28	0.35	3.75	–	1.18	1.75	8.70	–	-88.1	68.8	31.5
631	M10	0.89	0.25	0.33	4.13	–	2.00	–	8.13	–	-110.1	44.0	32.1
632	M7	1.01	0.28	0.38	3.75	–	2.00	1.75	8.70	–	-173.4	-7.4	-22.8
640	M30	0.80	0.28	0.33	3.88	–	1.20	1.80	8.38	5.00	-69.4	82.3	68.9
641	M34	0.89	0.28	0.33	3.75	–	2.10	1.75	8.48	8.25	-111.9	45.0	62.4
644	M33	0.89	0.28	0.33	3.75	–	1.18	1.70	9.50	8.25	-133.4	37.4	51.6
645	M42	1.08	0.25	0.25	3.75	–	1.15	1.50	9.50	8.00	-222.4	-42.1	-2.7
646	M43	1.20	0.30	0.40	3.88	–	1.63	2.63	8.00	8.25	-251.7	-84.7	-39.4
647	M46	1.26	0.30	0.48	3.95	–	3.15	2.05	8.25	8.30	-286.5	-112.8	-55.9
648	M47	1.20	0.28	0.33	3.75	–	1.25	1.55	9.63	5.00	-283.0	-94.7	-59.3
650	M2	0.83	0.28	0.33	4.13	–	1.98	6.13	5.00	–	-16.9	91.9	15.8
651	M3	1.05	0.28	0.33	4.13	–	2.50	5.88	5.63	–	-134.4	-5.9	-54.4
652	M3	1.20	0.28	0.33	4.13	–	3.00	5.88	4.88	–	-189.7	-63.7	-91.1
653	M4	1.33	0.28	0.33	4.25	–	4.13	5.88	4.65	–	-248.6	-118.4	-130.2
660	M35	0.83	0.25	0.25	4.15	–	1.95	6.40	5.00	5.00	-17.2	92.6	42.2
661	M36	0.85	0.28	0.33	4.13	–	2.00	3.00	5.00	8.25	-26.3	83.5	57.4
662	M6	0.80	0.28	0.33	4.13	–	1.50	4.25	5.00	12.00	-2.7	104.6	112.9
663	M15	1.55	0.25	0.25	4.38	–	5.00	6.50	4.00	5.00	-340.4	-207.2	-168.9
665	M41	1.10	0.28	0.25	4.25	–	2.00	6.75	3.75	5.00	-120.6	-14.4	-32.0
666	M44	1.15	0.33	0.33	4.25	–	2.00	5.25	6.25	12.00	-198.5	-55.8	-21.5
667	M45	1.25	0.33	0.33	4.25	–	1.60	8.00	5.00	5.50	-219.6	-88.7	-104.5

Remainder: Compositions calculated as in the middle of the ranges

S & H - Ms (C) = 561 - 474 (%C) - 30.4 (%Mn) - 17 (%Ni) - 17 (%Cr) - 21 (%Mo)

Andrews - Ms (C) = 539 - 423 (%C) - 30.4 (%Mn) - 17.7 (%Ni) - 12.1 (%Cr) - 7.2 (%Mo)

Cohen - Ms (C) = 493 - 300 (%C) - 33.3 (%Mn) - 16.6 (%Ni) - 22.2 (%Cr) - 11.1 (%Mo) - 11.1 (%W) - 5.6 (%Co) + 16.6 (%Al)

Table 6. cont'd

Alloy		Nominal composition (%)									Ms Temperature (°C)		
No	Name	C	Mn	Si	Cr	Ni	V	W	Mo	Co	S & H	Andrews	Cohen
110	W1	1.00	0.25	0.25							78.8	108.4	181.9
120	W2	1.00	0.25	0.25			0.25				78.8	108.4	181.9
122	W3	0.90	0.40	0.55			0.11				121.2	146.1	203.6
131	W4	1.00	0.25	0.25	0.23						74.8	105.6	176.8
132	W5	1.08	0.25	0.25	0.50						32.3	68.5	146.8
140	W7	1.01	0.25	0.25	0.35		0.20				72.8	104.2	174.1
310	S2	0.48	0.40	1.05			0.23		0.45		310.8	320.4	319.0
312	S4	0.58	0.78	2	0.25		0.23				256.4	267.2	265.7
313	S5	0.58	0.80	2.00	0.28		0.23		0.78		238.5	290.1	255.3
314	S6	0.45	1.35	2.25	1.35		0.30		0.40		271.8	288.3	253.7
320	S1	0.48	0.23	0.68	1.40		0.23	2.25			302.1	312.0	277.7
324	S3	0.50	0.25	0.25	0.95			1.00			299.6	308.4	299.7
393	O6	1.40	0.70	1.03	0.20				0.25		-134.3	-78.8	31.0
395	A10	1.38	1.85	1.25		1.80			1.50		-216.3	-144.1	-43.0
410	O1	0.93	1.20	0.25	0.50		0.23	0.50			72.1	103.1	1154.6
411	O2	0.90	1.60	0.25	0.23		0.18	1.50	0.25		72.4	105.0	142.4
413	O7	1.20	0.25	0.25	0.60		0.23	1.50	0.25		-31.5	14.7	89.2
420	A2	1.00	0.63	0.25	5.13		0.33		1.25		-47.3	25.4	41.5
422	A3	1.25	0.50	0.25	5.13		1.10		1.15		-159.4	-75.6	-28.1
423	A5	1.00	3.00	0.25	1.05				1.15		-54.0	3.5	54.3
424	A4	1.00	2.00	0.25	1.55				1.15		-29.5	27.8	76.5
426	A6	0.70	1.65	0.25	1.05				1.30		129.6	170.3	187.5
427	A8	0.55	0.30	0.93	5.13		0.40	1.25	1.40		173.8	224.7	164.4
428	A9	0.50	0.30	1.05	5.13	1.50	1.10		1.55		168.8	218.1	165.4
430	D2	1.50	0.30	0.25	12.00		0.60		0.95		-373.9	-256.9	-246.7
431	D3	2.23	0.30	0.25	12.00		0.50		0.95		-729.9	-565.7	-46537
432	D3	2.18	0.30	0.25	12.00	0.50	0.60				-694.7	-546.3	-44835
433	D6	2.10	0.30	0.85	12.00			0.75			-648.3	-503.6	-431.2
434	D5	1.50	0.30	0.50	12.00	0.33	0.50		0.95	2.75	-389.5	-262.8	-239.6
435	D1	1.00	0.30	0.25	12.00		0.65		0.80		-143.7	-44.3	-95.0
440	A7	2.43	0.55	0.55	5.38		4.53	1	1.15		-724.6	-579.3	-403.7
442	D7	2.33	0.40	0.40	12.50		4.20		0.95		-789.1	-617.1	-511.8
513	S7	0.50	0.50	0.60	3.25				1.55		219.7	261.4	230.3
520	H11	0.38	0.35	1.00	5.13		0.45		1.35		253.8	295.4	227.4
521	H13	0.39	0.35	1.00	5.13		1.00		1.43		247.4	290.6	223.5
522	H12	0.35	0.35	1.00	5.13		0.35	1.35	1.50		264.8	307.0	219.7
524	H10	0.40	0.48	1.00	3.38		0.50		2.50		245.6	295.6	243.1
530	H14	0.4	0.35	1	5.13		0.25	4.63	0.15	0.5	269.5	296.0	186.1
531	H19	0.4	0.35	0.35	4.38		1.98	4.13	0.43	4.25	276.4	302.9	233.4
535	H16	0.55	0.60	0.90	7.25			7.25			157.2	200.4	56.6
536	H23	0.30	0.28	0.38	11.88		1.00	11.88			207.6	259.8	-6.1
540	H21	0.30	0.28	0.33	3.38		0.45	9.25			352.1	632.7	212.3
541	H20	0.30	0.25	0.45	2.00		5.00	9.50			376.6	380.3	239.8
543	H22	0.35	0.38	0.28	2.75		0.38	11.88			335.8	346.1	179.3
545	H25	0.25	0.28	0.28	4.13		0.70	15.00			363.1	374.8	147.4
547	H24	0.50	0.25	0.25	3.13		0.55	14.75			262.5	282.0	98.7

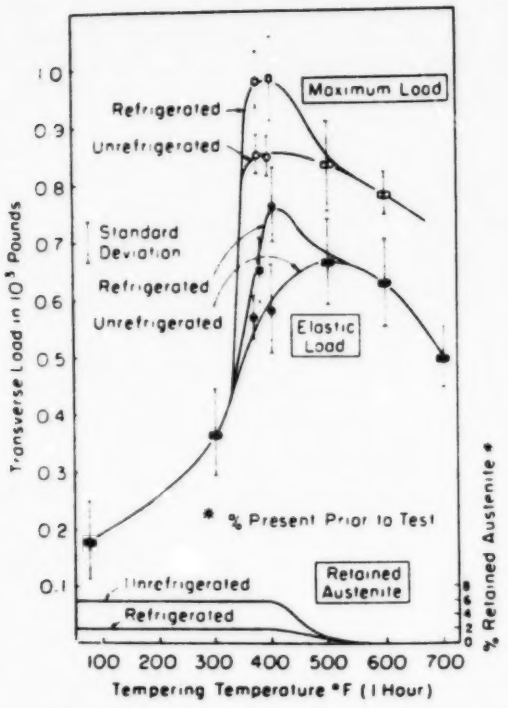


Fig. 1. Load versus tempering temperature for AISI 2340 steel. Slow-bend notched bar specimens tested at room temperature (12). (neg 20713)

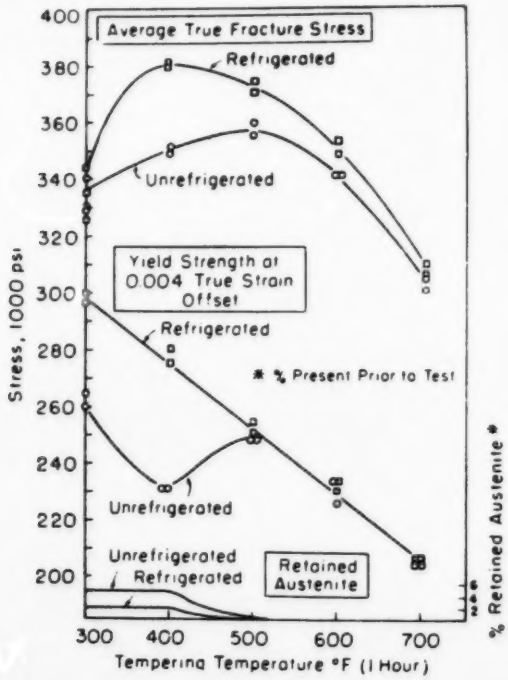


Fig. 2. Yield and fracture stress versus tempering temperature for AISI 2340 steel. Tensile specimens tested at room temperature (12). (neg 20715)

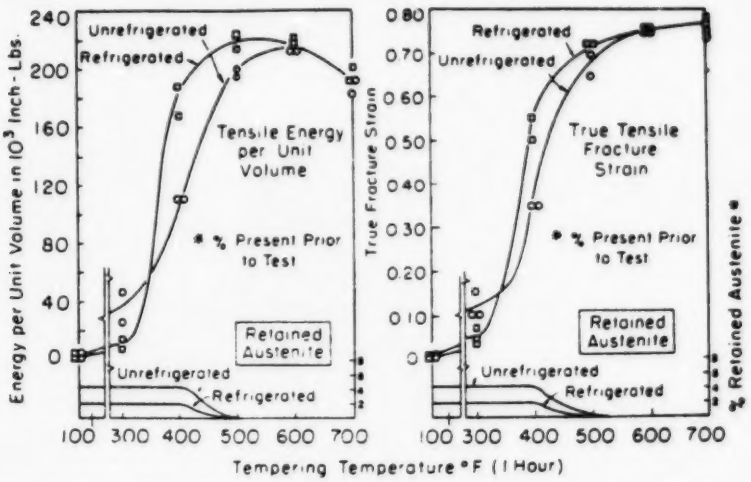
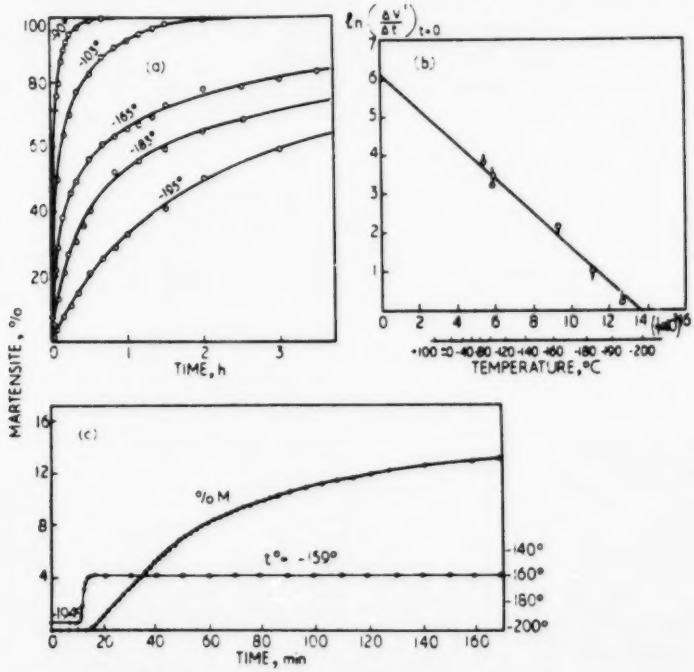


Fig. 3. Energy per unit volume and true fracture strain versus tmpering temperature for AISI 2340 steel. Tensile specimens tested at room temperature (12). (neg 20714)



(a) Transformation at different temperatures.

(b) Logarithm of initial rate of A--M transformation versus the reciprocal of the absolute temperature.

(c) Transformation at -159°C after supercooling of austenite in liquid nitrogen.

Fig. 4. Isothermal austenite-martensite transformation in a steel with 0.7% C, 6.5% Mn and 2% Cu (49). (neg 20712)